

SPECIFICATION

HIGH SILICON STAINLESS STEEL,

SPRING MADE THEREOF, AND

PROCESS FOR MANUFACTURING HIGH SILICON STAINLESS STEEL

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TECHNICAL FIELD

[0001] The present invention relates to a high silicon stainless steel. In particular, the invention relates to a high silicon stainless steel with high ductility, a spring made thereof, and a process for manufacturing the high silicon stainless steel.

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BACKGROUND ART

[0002] High silicon stainless steel is known by the name "silicolloy", a stainless material containing silicon in 3.5% by weight or more. As a metal material, high silicon stainless steel is endowed with excellent toughness, and is resistant to corrosion, wear and heat.

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[0003] On the other hand, high silicon stainless steel has an elongation at break of about 10% after forging and quenching. Additionally, in order to enhance hardness, high silicon stainless steel may be subjected to thermal aging at about 500°C. After the thermal aging, the elongation at break goes down to as little as 3.5%. Due to lack of ductility, which is one

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of the most characteristic properties for metal materials, high silicon stainless steel has limited applications to mechanical parts.

[0004] Regarding steel materials such as stainless steels, it is generally known that mechanical strength and ductility can be enhanced by making their grain size smaller. To reduce the grain size of steel materials such as stainless steels, some processes have been disclosed (e.g. Patent Documents 1-3).

10 [Patent Document 1] Japanese Patent Laid-open Publication No. 2000-248329

[Patent Document 2] Japanese Patent Laid-open Publication No. 2000-351040

[Patent Document 3] Japanese Patent Laid-open
15 Publication No.2002-192201

DISCLOSURE OF THE INVENTION

PROBLEM TO BE SOLVED BY THE INVENTION

[0005] However, in the case of high silicon stainless steel, if the processes as disclosed in Patent Documents 1-3 are applied in an attempt to refine the grains, these processes end with breaking the material or with other adverse results. Thus, it has been impossible to provide a high silicon stainless steel
20 with a refined grain structure. Specifically, the
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conventional processes provide a high silicon stainless steel with a grain size of about 25 to 30 μm at smallest. As mentioned already, its elongation at break is about 10% after forging and quenching, and as little as 3.5% after thermal aging.

[0006] By solving such a problem and realizing a high silicon stainless steel with a high elongation at break and remarkable ductility, it is possible to take further advantage of the properties of high silicon stainless steel and to provide high-quality mechanical parts, etc. In view of this, there has been a strong demand for a high silicon stainless steel with a high elongation at break.

[0007] Being made in light of this situation, the present invention aims to provide a high silicon stainless steel with a high elongation at break, a spring made thereof, and a process for manufacturing the high silicon stainless steel.

20 MEANS TO SOLVE THE PROBLEMS

[0008]

<Summary of the Invention>

The present invention derives from discovery of a grain refinement process in high silicon stainless steel. To be specific, the present invention can reduce

the grain size of a high silicon stainless steel by forging the high silicon stainless steel or its master alloy under impact load and/or static load, preferably under impact load, while regulating the surface temperature of the high silicon stainless steel or the master alloy within a certain range. Besides, the present invention can control the grain size by changing the surface temperature condition and the forging condition.

10 **[0009]**

<Solutions>

The high silicon stainless steel of the present invention is characterized in mainly comprising a microstructure with a grain size of 15 μm or less, and having an elongation at break of 12% or higher.

[0010] With a grain size of 15 μm or less, the high silicon stainless steel can achieve a higher elongation at break.

[0011] Preferably, the high silicon stainless steel of the present invention mainly comprises a microstructure with a grain size of 7 μm or less, and has an elongation at break of 14% or higher.

[0012] With a grain size of 7 μm or less, the high silicon stainless steel can achieve a still higher elongation at break.

[0013] In this context, the high silicon stainless steel is a stainless steel containing Si in 3.5% by weight or more, generally from 3.5 to 7% by weight, as typically represented by Silicolloy A1, Silicolloy A2 and
5 Silicolloy D.

[0014] The term "grain size" as used herein means a value obtained according to ASTM Designation E112-82. The term "elongation at break" as used herein refers to the one defined in JIS Z2241, Method of tensile test
10 for metallic materials.

[0015] A spring made of the high silicon stainless steel of the invention shows a dramatic improvement in ductility. As a consequence, the spring is less likely to break even under a heavy load and can be an excellent
15 mechanical part or the like. In addition, the spring itself has a long life.

[0016] The high silicon stainless steel of the present invention is further characterized in that any of the high silicon stainless steels mentioned above
20 is subjected to thermal aging at a temperature range of 480 to 550°C, and has an elongation at break of 7% or higher after the thermal aging. In many cases, thermal aging of the high silicon stainless steel is conducted within the above-mentioned temperature range
25 for about an hour.

[0017] The thermal aging increases hardness at the surface of the material. The high silicon stainless steel which went through the thermal aging and which has an elongation at break of 7% or higher can retain
5 a remarkable Brinell hardness of 450 or higher. The term "Brinell hardness" as used herein means a value obtained according to JIS Z2243, Brinell hardness test.

[0018] The high silicon stainless steel with such a high hardness can provide excellent mechanical parts
10 or the like, including a highly durable, long-life spring.

[0019] The spring and other mechanical parts may be subjected to surface treatment such as nitriding and/or shot peening. In general, surface hardness of
15 high silicon stainless steel increases when nitrogen is allowed to diffuse into the surface. For the high silicon stainless steel with a refined grain structure, nitriding can increase the surface hardness still further. Additionally, shot peening causes generation
20 of residual stress inside high silicon stainless steel. With respect to the high silicon stainless steel which has a refined grain structure and whose surface hardness has increased by nitriding, shot peening assists generation of a greater residual stress, making the high
25 silicon stainless steel resistant to a greater stress.

[0020] The process of the invention for manufacturing the high silicon stainless steel is characterized in comprising the step of forging a high silicon stainless steel or a master alloy thereof. The forging step includes: a load application step for applying an impact load and/or a static load to the high silicon stainless steel or the master alloy, wherein a surface temperature of the high silicon stainless steel or the master alloy is kept at 1,100°C or higher, and is later dropped to a temperature range of 950°C or below and not so low as to break the high silicon stainless steel or the master alloy. The process provides a steel material which mainly comprises a microstructure with a grain size of 15 μm or less.

[0021] This process provides a high silicon stainless steel having a high elongation at break. In this process, application of a load starts at a surface temperature of 1,100°C or higher and continues until the surface temperature drops to 950°C or below, thereby promoting grain refinement in the high silicon stainless steel or its master alloy. As the forging time at 950°C or below is longer, the grain size becomes smaller. Further, within the temperature range which is 950°C or below and not so low as to break the high silicon stainless steel or the master alloy, load application

at a lower temperature promotes further grain refinement. At the start of forging, the surface temperature of the high silicon stainless steel or its master alloy is preferably between 1,100 and 1,200°C because the temperature does not need to be above 1,200°C. When the surface temperature is lower than 1,100°C at the start of forging, the high silicon stainless steel or its master alloy has not yet gained sufficient ductility and is more likely to break. In this context, the term "master alloy" means an alloy composed of a material which becomes a high silicon stainless steel after the forging.

[0022] The load to be applied during the forging may be a static load or an impact load. However, application of an impact load induces active self-heating inside the high silicon stainless steel or its master alloy, thereby further promoting grain refinement and saving the time required for the forging step. Impact load may be combined with static load. For example, application of an impact load may be followed by rolling (application of an static load), which facilitates manufacture of a thin plate-shaped material.

[0023] The process of the invention for manufacturing the high silicon stainless steel is char-

characterized in comprising: a first load application step for applying an impact load and/or a static load to the high silicon stainless steel or the master alloy, wherein a surface temperature of the high silicon stainless steel or the master alloy is kept at 1,100°C or higher, and is later dropped to a temperature range of 950°C or below and not so low as to break the high silicon stainless steel or the master alloy; and a second load application step for applying an impact load and/or a static load to the high silicon stainless steel or the master alloy, wherein a surface temperature of the high silicon stainless steel or the master alloy is kept at a temperature range from 850 to 1,050°C, and is later changed to a temperature range of 950°C or below and not so low as to break the high silicon stainless steel or the master alloy. The first load application step is followed by the second load application step once or more. The process provides a steel material which mainly comprises a microstructure with a grain size of 15 μm or less.

[0024] As described earlier, a high silicon stainless steel with a refined grain structure is obtained by application of a load to the high silicon stainless steel or the master alloy, with the surface temperature being maintained in a temperature range of

950°C or below and not so low as to break the high silicon stainless steel or the master alloy. Moreover, by combining the first load application step and the second load application step, it is easier to avoid break of the high silicon stainless steel or the master alloy during the forging. At the start of the second load application step, the surface temperature of the high silicon stainless steel or the master alloy is regulated to not higher than 1,050°C. If the surface temperature exceeds 1,050°C under heating, the grain size becomes larger again. The second load application step may be performed only once or more than once.

[0025] The process of the invention for manufacturing the high silicon stainless steel is characterized in that: a lowest surface temperature for the second load application step is lower than a lowest surface temperature for the first load application step; the second load application step is conducted more than once, during which a lowest surface temperature for each second load application step is lower than a lowest surface temperature for a previous second load application step so as to reduce a grain size little by little; and the grain size is controlled by changing the number of times for conducting the second load application step. The production process provides a

steel material which mainly comprises a microstructure with a grain size of 15 μm or less.

[0026] This process can provide a high silicon stainless steel which has a high elongation at break
5 and can control the grain size.

[0027] As described above, the grain size is reduced little by little, by gradually lowering the lowest surface temperature for each load application step. In other words, ductility of the high silicon stainless
10 steel or its master alloy increases little by little, so that the high silicon stainless steel or its master alloy is less likely to break. Eventually, the grain size can be reduced every time the load application step is repeated.

15 [0028] Even when the lowest temperature for each load application step is not gradually lowered, the grain size becomes smaller every time the load application step is repeated. In this case, to prevent break of the high silicon stainless steel or its master alloy,
20 it is preferable to apply a smaller amount of load during earlier load application step.

EFFECTS OF THE INVENTION

[0029] By reducing the grain size to 15 μm or less,
25 the invention provides a high silicon stainless steel

which achieves an improved elongation at break and excellent ductility. Further, by reducing the grain size to 7 μ m or less, the invention provides a high silicon stainless steel whose elongation at break is dramatically improved to 14% or higher.

[0030] With respect to a high silicon stainless steel whose hardness has increased by the thermal aging, the invention ensures an elongation at break of as high as 7% or more. This is an outstanding improvement, as understood from a comparison with a conventional value. The high silicon stainless steel achieves not only an elongation at break of 7% or higher but also a Brinell hardness or 450.

[0031] A spring made of the high silicon stainless steel shows an outstanding improvement in ductility. This spring is unlikely to break even under a heavy load and has a long life.

[0032] The process of the invention for manufacturing the high silicon stainless steel can reduce its grain size to 15 μ m or less.

BRIEF DESCRIPTION OF DRAWINGS

[0033] Fig. 1 schematically shows a manner of forging a high silicon stainless steel, according to an Example of the present invention. Fig. 1(a) il-

lustrates how the forging is performed. Fig. 1(b) is an external perspective view of the high silicon stainless steel.

Fig. 2 concerns electron microscopic observation of the structure of the high silicon stainless steel, according to Example 1 of the present invention. Fig. 2(a) is a schematic illustration of the observation area. Fig. 2(b) is a photographic image of the peripheral structure, and Fig. 2(c) is a photographic image of the central structure.

Fig. 3 illustrates disc springs made of the high silicon stainless steel, according to Example 2 of the present invention. Fig. 3(a) is a front sectional view of a washer constituting a stack of disc springs. Fig. 3(b) is a front sectional view of a stack of disc springs.

Fig. 4 concerns electron microscopic observation of the structure of a conventional high silicon stainless steel. Fig. 4(a) is a schematic illustration of the observation area. Fig. 4(b) is a photographic image of the peripheral structure, and Fig. 4(c) is a photographic image of the central structure.

DESCRIPTION OF THE NUMERALS

[0034]	1	master alloy
25	2	air hammer

3 anvil
4 hammer
5 driving mechanism
6 thermometer
5 7 operator
8 gripper
101 high silicon stainless steel
31 washer
32 disc spring

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BEST MODE FOR CARRYING OUT THE INVENTION

[0035] An embodiment of the present invention is hereinafter described.

[0036] An embodiment of the present invention
15 encompasses a high silicon stainless steel which is mainly composed of a microstructure with a grain size of 15 μm or less and which has an elongation at break of 12% or higher; and a high silicon stainless steel which is mainly composed of a microstructure with a grain
20 size of 7 μm or less and which has an elongation at break of 14% or higher.

[0037] The high silicon stainless steel is widely used as a material for metal products such as mechanical parts. By way of example, a spring made of the high
25 silicon stainless steel is resistant to corrosion and

has a long life, unlike conventional springs.

[0038] The high silicon stainless steel according to the embodiment achieves an elongation at break of 7% or higher, after thermal aging at a temperature range of 480 to 550°C. In addition, the high silicon stainless steel which went through the thermal aging can be endowed with a Brinell hardness of 450 or higher while keeping an elongation at break of 7% or higher. The high silicon stainless steel having such a high hardness can provide a highly durable, long-life spring.

[0039] Refinement of the structure of the high silicon stainless steel is effected to a material for a high silicon stainless steel, or a master alloy composed of a material which becomes a high silicon stainless steel (hereinafter, the material and the master alloy before grain refinement are generally called "master alloy or the like"). The size and shape of the master alloy or the like are not particularly limited. Depending on the manufacturing facilities and purpose, the master alloy or the like may be in various sizes and may be round, block-shaped, plate-shaped or shaped otherwise. It goes without saying that the high silicon stainless steel can be processed into a round, block, plate or other shape of various sizes, through the manufacturing process including forging and the

others.

[0040] As detailed later, the high silicon stainless steel is manufactured while a load is applied to the master alloy or the like within a given temperature range. The load may be either an impact load or a static load, of which an impact load is preferred because it accelerates progress of the grain refinement. Typically, a device for applying an impact load may be a hammer-equipped press machine.

10 **[0041]** In order to obtain a high silicon stainless steel having a refined grain structure, the master alloy or the like is subjected to forging at a temperature of 950°C or below, thereby refining grains in the master alloy or the like.

15 **[0042]** To start the manufacturing process, load is applied to the master alloy or the like which has been heated to have a surface temperature of 1,100 to 1,200°C. Due to exposure to external air, the temperature of the master alloy or the like drops while the load is applied.

20 In due course, the surface temperature of the master alloy or the like reaches 950°C or below, but even then the load application is continued. Preferably, the surface temperature of the master alloy or the like is reduced to as low as possible, but not so low as to break

25 the master alloy or the like. It should be borne in

mind that the master alloy or the like tends to break at 700°C or below.

[0043] For further grain refinement, load is applied for as long as possible, with the temperature
5 being kept at 950°C or below, preferably 850°C or below. During this forging, the temperature is allowed to drop to a lowest possible temperature at which the master alloy or the like does not break.

[0044] After the end of the load application, the
10 master alloy or the like is cooled by quenching in a conventional manner, thereby giving a high silicon stainless steel with a refined grain structure.

[0045] The high silicon stainless steel according to the embodiment of the invention is manufactured in
15 the above-mentioned manner. Additionally, by performing the load application (forging) step more than once as described below, it is easier to control the grain size of the high silicon stainless steel between 0.6 and 15 μm .

[0046] To start with, a load is applied to the master
20 alloy or the like which is heated to near 1,100 to 1,200°C. The forging is stopped when the temperature drops to a temperature range of 950°C or below and not so low as to break the master alloy or the like (first forging
25 step).

[0047] Next, the master alloy or the like is heated until its surface temperature reaches 850°C or higher, preferably near 1,050°C. The surface temperature should not exceed 1,050°C because such a high temperature allows the grain size to get larger. Then, a load is applied again to the master alloy or the like whose surface temperature is near 1,050°C. The forging is stopped when the temperature drops to a temperature range of 950°C or below, preferably 850°C or below, and not so low as to break the master alloy or the like (second forging step). Once again, the master alloy or the like is heated until its surface temperature reaches near 1,050°C. Then, a load is applied again to the master alloy or the like whose surface temperature is near 1,050°C until the temperature drops to a temperature range of 950°C or below and not so low as to break the master alloy or the like (third forging step). Where necessary, the forth, fifth and more forging steps may be repeated.

20 [0048] In order to facilitate reduction of the grain size, the temperature for stopping the second forging step is set lower than the one for stopping the first forging step. By gradually lowering the lowest temperature for each forging step, it is possible to apply a heavy load while avoiding break of the master

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alloy, and eventually to obtain refined grains easily.

[0049] As described, the grain size is reduced little by little while the above-mentioned forging step is repeated. Hence, it is possible to control the grain size by setting the number of times for conducting the forging step, depending on a desired grain size. In other words, the grain size of a microstructure can be controlled more easily if the forging step is made up of more than one forging steps.

10 **[0050]** Similar to the foregoing description, the last step in the manufacturing process is to cool the master alloy or the like by quenching in a conventional manner. Thus obtained is a high silicon stainless steel according to the embodiment.

15 **[0051]** Now, referring to the drawings, the present invention is specifically described by way of Examples. These Examples are given merely for the purpose of description and should not be construed as limiting the invention.

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EXAMPLE 1

[0052] A master alloy used in this Example had a diameter of 12 cm and a length of 25 cm. The composition of its main components, except Fe (iron), was Si:4, C:0.02, Ni:7, Cr:12 (unit: % by weight). This master

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alloy was subjected to forging and quenching in the manner mentioned below. Thus obtained was a high silicon stainless steel which had a diameter of 3 cm and a length of 120 cm.

5 **[0053]** Fig. 1 schematically shows how to forge a high silicon stainless steel according to an Example of the present invention. Fig. 1(a) illustrates how the forging is performed. Fig. 1(b) is an external perspective view of the high silicon stainless steel
10 thus obtained.

[0054] To start with, the master alloy 1 heated to 1,150°C was placed on an anvil 3 of a 0.5-ton air hammer 2.

[0055] For forging, a hammer 4 was allowed to fall
15 from 70 cm above the anvil 3 onto the master alloy 1. To apply the impact, a driving mechanism 5 let the hammer 4 fall and rise at a cycle of twice per second. An operator 7 moved the master alloy 1 properly so as to forge the entirety of the master alloy 1.

20 **[0056]** The surface temperature of the master alloy 1 was monitored by a thermometer 6. When the surface temperature dropped to 850°C, the forging was stopped. Then, the master alloy 1 was put into an electric furnace (not shown) and heated until its surface temperature
25 rose to around, but not exceeding, 1,050°C. During this

heating, the surface temperature of the master alloy 1 was also monitored by the thermometer 6. As the thermometer 6, a digital radiation thermometer (produced by Daido Steel Co., Ltd.; Starthermo DS-06CF) was employed.

[0057] Next, the master alloy 1 heated up to near 1,050°C was forged again in the same manner as above. At this stage, the master alloy 1 was forged until its surface temperature dropped to 800°C. Then, the master alloy 1 was put into the electric furnace and heated until its surface temperature rose to 1,000°C.

[0058] The master alloy 1 heated up to near 1,000°C was forged once again in the same manner as above. At this stage, the master alloy 1 was forged until its surface temperature dropped to 750°C. Then, a series of forging steps were terminated.

[0059] After a series of forging steps, the master alloy 1 was heated in the electric furnace until its surface temperature reached 1,000°C. Thereafter, the master alloy 1 was subjected to water quenching (generally called ST treatment) to give a high silicon stainless steel 101.

[0060] With respect to this high silicon stainless steel 101, the tensile strength was 1,134 N/mm² and the elongation at break was 14%. The Brinell hardness was

341.

[0061] Additionally, the high silicon stainless steel 101 was subjected to thermal aging at 500°C for one hour. With respect to the high silicon stainless steel which went through the thermal aging, the tensile strength was 1,634 N/mm² and the elongation at break was 10%. The Brinell hardness was 461.

[0062] For both evaluations, test pieces were prepared according to JIS Z2201, Test pieces for tensile test for metallic materials (test piece No. 14A4). The tensile strength and the elongation at break were measured by the tensile test according to JIS Z2241, Method of tensile test for metallic materials. The Brinell hardness was measured according to JIS Z2243.

[0063] Regarding the high silicon stainless steel 101 which went through the thermal aging, its cross-section was observed at a part near the external circumference (the periphery) and at a part near the center (the center). The grain size was measured according to ASTM Designation E112-82.

[0064] Fig. 2(a) is a schematic cross-sectional view of the observation area. Figs. 2(b) and 2(c) are photographic images at the periphery and the center, respectively, showing the microstructure of the high silicon stainless steel which went through the thermal

aging. These photographic images were taken by an electron microscope (magnification x 400). As indicated by comparison between Figs. 2(b) and 2(c), there is no difference between the peripheral structure and the central structure. The grain size was 6.9 μm at both the periphery (see Fig. 2(b)) and the center (see Fig. 2(c)).

[0065] It should be understood that the grain size is not affected by the thermal aging and remains unchanged before and after the thermal aging.

[0066] Additionally, the high silicon stainless steel which went through the thermal aging was subjected to conventional nitriding and conventional shot peening (conventional airless shot peening). After these treatments, the Vickers hardness of the high silicon stainless steel was 1,400 at the surface. The hardness was evaluated by the Vickers hardness test according to JIS Z2244.

[0067] Furthermore, the high silicon stainless steel according to Example 1 was compared with a conventional high silicon stainless steel. Using a sample of a commercial (conventional) high silicon stainless steel, the surface of the sample was observed and its photographic images were taken in the above-mentioned manner. The grain size was also

measured in the above-mentioned manner.

[0068] Fig. 4(a) is a schematic cross-sectional view of the observation area, regarding the sample of the conventional high silicon stainless steel. Figs. 4(b) and 4(c) are photographic images at the periphery and the center, respectively, showing microstructures of the same which went through the thermal aging. These photographic images were taken by an electron microscope (magnification $\times 400$). The photographic images did not reveal any significant difference between the peripheral structure and the central structure (Figs. 4(b) and 4(c)), except a slight difference in grain size. The grain size of the conventional high silicon stainless steel was $27.2\text{ }\mu\text{m}$ at the periphery (see Fig. 4(b)) and $24.9\text{ }\mu\text{m}$ at the center (see Fig. 4(c)).

EXAMPLE 2

[0069] Disc springs were manufactured using the high silicon stainless steel 101 obtained in Example 1 (diameter 3 cm, length 120 cm).

[0070] As the disc springs, washers for constituting disc springs were prepared and stacked on top of each other.

[0071] Fig. 3(a) is a front sectional view of a washer. Fig. 3(b) is a front sectional view of a stack

of disc springs.

[0072] To manufacture the washer 31 shown in Fig. 3(a), the high silicon stainless steel 101 (see Fig. 1) was cut into columnar materials, each having a diameter of 3 cm (30 mm) and a length of 10 cm (100 mm). The bottom of each columnar material was struck so as to enlarge its diameter to about 40 mm. Then, each columnar material was sliced to give discs each having a diameter of about 40 mm and a thickness of about 2.5 mm. Each of these discs was perforated at the center so as to have a hole with a diameter of about 20 mm, and the edge was rounded. Thus obtained was a perforated disc-like material.

[0073] The perforated disc-like material was made to curve under stress and to assume a substantially horn-like shape with the central part protruded. This material was subjected to thermal aging at 500°C for one hour, thereby making a washer 31. The main dimensions of the washer 31 are given in Fig. 3(a).

[0074] Next, 130 pieces of washers 31 were stacked on top of each other as illustrated in Fig. 3(b), thereby forming a stack of disc springs 32.

[0075] A stack of disc springs 32 was subjected to a life test under vertical load (in the directions of Arrows a and b). Using a servopulser tester, load was

applied at a cycle of 10 times per second (10 Hz). The amplitude was from 4.5 to 3.2 kN. Even after the load was applied 8 million times, the disc springs 32 suffered from no particular damage.

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INDUSTRIAL APPLICABILITY

[0076] The high silicon stainless steel of this invention is utilized not only for springs, but also for a wide variety of metal products. In particular, it is applicable to metal products which require high strength and high toughness, such as mechanical parts (bearings, bolts and nuts, etc.), structural members (roller bearings, etc.), cutleries, cutting tools, and more.

15 [0077] The process of this invention for manufacturing the high silicon stainless steel is assumed to be applicable to other metals than high silicon stainless steel and achieve grain refinement of their structures, as far as the precipitation hardening stainless steels are concerned. Such precipitation
20 hardening stainless steels include, for example, SUS 630.